Application No. 09/486,343 Attorney's Docket No. 004900-174 Page 2

CI

conducting a one-step emulsion-and-blocking reaction by placing an isocyanate composition comprising free isocyanate functions in contact with at least one blocking agent A in the presence of a surfactant B and an aqueous phase, the isocyanate composition being added gradually to a stock containing at least some of the aqueous phase and at least some of the blocking agent so that the content of free isocyanate functions is equal to not more than 2 equivalents per kg in the isocyanate phase.

- 67. (New) The process according to claim 66, wherein the composition containing an isocyanate function contains, on average, 1 to 5 isocyanate functions per molecule bearing isocyanate function(s).
- 68. (New) The process according to claim 66, wherein the composition containing an isocyanate function contains, on average, 4/3 to 4 isocyanate functions per molecule bearing isocyanate function(s).
- 69. (New) The process according to claim 66, wherein the blocking agent contains at least one labile hydrogen.
- 70. (New) The process according to claim 66, wherein the blocking agent contains at least one labile hydrogen and wherein the pKa of the reactive hydrogens is at least equal to 4 but is not more than 14, with the exception of lactams.

Application No. <u>09/486,343</u> Attorney's Docket No. <u>004900-174</u> Page 3

71. (New) The process according to claim 66, wherein the pKa of the reactive hydrogens is equal to 5.

- 72. (New) The process according to claim 66, wherein the pKa of the reactive hydrogens is equal to 6.
- 73. (New) The process according to claim 66, wherein the pKa is not ore than 13.
- 74. New) The process according to claim 66, wherein the pKa is not more than 12.
- 75. (New) The process according to claim 66, wherein the pKa is not more than 10.
- 76. (New) The process according to claim 66, wherein the pH of the aqueous phase is maintained at a value of not more than 12 throughout the reaction.
- 77. (New) The process according to claim 66, wherein the pH of the aqueous phase is maintained at a value of not more than 11 throughout the reaction.

Coup.

Application No. <u>09/486,343</u> Attorney's Docket No. <u>004900-174</u> Page 4

78. (New) The process according to claim 66, wherein the pH of the aqueous phase is maintained at a value of not more than 10 throughout the reaction.

79. (New) The process according to claim 66, wherein the pH of the aqueous phase is maintained at a value at least equal to the value (pKa-2) throughout the reaction.

80. (New) The process according to claim 66, wherein the pH of the aqueous phase is maintained at a value at least equal to the value (pKa-1) throughout the reaction.

- 81. (New) The process according to claim 66, wherein the pH of the aqueous phase is maintained at a value at least equal to the pKa value of the blocking agent, or one of the blocking agents, throughout the reaction.
- 82. (New) The process according to claim 66, wherein the reaction is carried out at a temperature not greater than the cloud point temperature of the surfactant or of a mixture of surfactants used.
- 83. (New) The process according to claim 66, wherein the composition containing an isocyanate function, contains a solvent, including a mixture of solvents.

Cont

84. (New) The process according to claim 66, wherein said step of placing the composition in contact is carried out by stirring with a mixer under conditions which ensure that at least 90% by mass of the particles are between 0.005 and 50 micrometers in size.

Cont.

- 85. (New) The process according to claim 83, wherein 95% by mass of the particles are between 0.005 and 50 micrometers in size.
- 86. (New) The process according to claim 83, wherein the stirring is carried out using a grinding mixer.
- 87. (New) The process according to claim 66, wherein the reaction mixture is subjected to recirculation.
- 88. (New) The process according to claim 66, wherein the reaction mixture is subjected to recirculation, during which it is subjected to the action of a grinding mixer.
- 89. (New) The process according to claim 66, wherein said step of placing the composition in contact is carried out by adding reagent(s) to a stock containing at least one aqueous phase and at least some of the blocking agent(s).

90. (New) The process according to claim 66, wherein said step of placing the composition in contact is carried out by adding reagent(s) to a feed stock containing at least one aqueous phase, at least some of the surfactant(s) and at least some of the blocking agent(s).

- Cont with
- 91. (New) The process according to claim 66, further comprising:
- b) subjecting the mixture obtained in a) to a shear (speed gradient) of greater than $1000 \, s^{-1}$ but less than $1,000,000 \, s^{-1}$; and
- c) repeating step b), optionally after step a) has been repeated, until a stable emulsion is obtained whose particles have a Sauter diameter of greater than 0.1 μ m but less than 5 μ m and a dispersion width of less than 5 μ m.
- 92. (New) The process according to claim 90, wherein the shear (speed gradient) is greater than 20,000s⁻¹.
- 93. (New) The process according to claim 90, wherein the shear (speed gradient) is less than 200,000s⁻¹.
- 94. (New) The process according to claim 90, wherein the particles have a Sauter diameter of greater than 2 μm .

Application No. 09/486,343 Attorney's Docket No. 004900-174 Page 7

95. (New) The process according to claim 90, wherein the particles have a Sauter diameter of less than 2 μm .

Cond

- 96. (New) The process according to claim 91, wherein step c) is continued after adding the isocyanate composition.
- 97. (New) The process according to claim 91, wherein step c) is carried out by recirculating the emulsion obtained after step b).
- 98. (New) The process according to claim 91, wherein the reaction mixture is recirculated in a colloidal mill.
- 99. (New) The process according to claim 98, wherein the aqueous phase, to which the isocyanate- composition, optionally containing at least one of the surfactant and the solvent is added, is subjected to a first shear (speed gradient) of less than 20,000 s⁻¹, after placing the composition in contact with the blocking agent and before the high-shear step b).
- 100. (New) The process according to claim 99, wherein the first shear (speed gradient) is less than $10,000 \text{ s}^{-1}$.

Application No. 09/486,343 Attorney's Docket No. 004900-174 Page 8

101. (New) The process according to claim 66, wherein the process is carried out at a temperature of less than 50° C.

Cong.

- 102. (New) The process according to claim 101, wherein the temperature is less than 40° C.
- 103. (New) The process according to claim 66, wherein all or some of the surfactant is added to the isocyanate composition in a proportion of less than 20% by weight relative to the weight of the isocyanate composition to be blocked.
- 104. (New) The process according to claim 103, wherein the surfactant is added in a proportion of less than 10% by weight relative to the weight of the isocyanate composition to be blocked.
- 105. (New) The process according to claim 66, wherein the surfactant is a surfactant containing an anionic function.
- 106. (New) The process according to claim 66, wherein the said surfactant is an anionic surfactant containing at least one function selected from the group consisting of an aryl phosphate or an alkyl phosphate, an aryl phosphonate, an alkyl phosphonate, and phosphinate.

Application No. <u>09/486,343</u> Attorney's Docket No. <u>004900-174</u> Page 9

107. (New) The process according to claim 66, wherein the process includes a compound containing an anionic function and at least one of a polyethylene glycol and polypropylene glycol chain fragment of at least 1 oxyethylene or oxypropylene units.

Cont

- 108. (New) The process according to claim 107, wherein the at least one of polyethylene glycol and polypropylene chain fragments comprise at least 5 oxyethylene or oxypropylene units.
- 109. (New) The process according to claim 107, wherein the at least one of polyethylene glycol and polypropylene chain fragments comprise at least 7 oxyethylene or oxypropylene units.
- 110. (New) The process according to claim 66, wherein the compound comprises a hydrophilic part formed of at least one of an anionic function, a polyethylene glycol chain fragment and a polypropylene glycol chain fragment, and a lipophilic part based on a hydrocarbon-based radical.
- 111. (New) The process according to claim 110, wherein the lipophilic part is selected from the group consisting of an alkyl group and an aryl group.

112. (New) The process according to claim 66, wherein the surfactant is an anionic surfactant and wherein an anionic part of the surfactant corresponds to the following formula:

$$(O)_{p} \xrightarrow{(X)_{p}} (X \xrightarrow{O \setminus s}_{R_{2}})_{q}$$

$$(O)_{p} \xrightarrow{E} (X \xrightarrow{O \setminus n}_{R_{1}})_{q}$$

where q represents zero or 1,

where p represents an integer between 1 and 2 (closed intervals, i.e. including the limits);

where m represents zero or an integer between 1 and 2 (closed intervals, i.e. including the limits);

where X and X', which are similar or different, represent a divalent radical containing not more than two carbon-based-chain members;

where s is zero or an integer chosen between 1 and 30 (closed intervals, i.e. including the limits);

where n is zero or an integer chosen between 1 and 30 (closed intervals, i.e. including the limits);

where E is phosphorus;

where R_1 and R_2 , which are similar or different, represent a hydrocarbon-based radical.

Cond.

113. (New) The process according to claim 112, wherein s is an integer between 5 and 25.

Colf.

- 114. (New) The process according to claim 112 wherein s is an integer between 9 and 20.
- 115. (New) The process according to claim 112, wherein n is an integer between 5 and 25.
- 116. (New) The process according to claim 112, wherein n is an integer between 9 and 20.
- 117. (New) The process according to claim 112, wherein the hydrocarbon-based radical is an optionally substituted aryl or an optionally substituted alkyl.
- 118. (New) A method of blocking isocyanates, the method comprising placing an (31) ionic surfactant in contact with at least one blocking and an aqueous phase in an emulsion, wherein the surfactant comprises a hydrophilic part containing an anionic function selected from the group consisting of a phosphate, a phosphonate and a phosphinate group, and at least one of a polyethylene glycol chain fragment and a

polypropylene glycol chain fragment and a lipophilic part selected from the group consisting of an alkyl group and an aryl group.

Cont

- 119. (New) The method according to claim 118, wherein the surfactant comprises a compound comprising an anionic function and at least one of a polyethylene glycol chain fragment and a polypropylene glycol chain fragment of at least 5 oxyethylene units.
- 120. (New) The method according to claim 119, wherein the at least one polyethylene glycol chain fragment and polypropylene glycol chain fragment comprise at least 7 oxyethylene units.
- 121. (New) The method according to claim 119, wherein the compound comprises a hydrophilic part formed of the anionic function, the polyethylene glycol chain fragment, and a lipophilic part based on a hydrocarbon-based radical.
- 122. (New) The method according to claim 121, wherein the lipophilic part is selected from the group consisting of an alkyl group and an aryl group.
- 123. (New) The method according to claim 118, wherein the anionic part of the surfactant corresponds to formula I below:

Application No. 09/486,343 Attorney's Docket No. 004900-174 Page 13

$$(O)_{p} \xrightarrow{(X - (O)_{p} - (X - (O)_{p})_{R_{1}}} R_{2})_{q}$$

where q represents zero or 1;

where p represents an integer between 1 and 2 (closed intervals, i.e. including the limits);

where m represents zero or an integer between 1 and 2 (closed intervals, i.e. including the limits);

where X and X', which are similar or different, represent a divalent radical containing not more than two carbon-based chain members;

where s is zero or an integer chosen between 1 and 30 (closed intervals, i.e. including the limits);

where n is zero or an integer chosen between 1 and 30 (closed intervals, i.e. including the limits);

where E is phosphorus; and

where R_1 and R_2 , which are similar or different, represent a hydrocarbon-based radical.

124. (New) The method according to claim 123, wherein s is an integer between 5 and 25.

Cont

125. (New) The method according to claim 123, wherein s is an integer between 9 and 20.

Cont.

- 126. (New) The method according to claim 123, wherein n is an integer between 5 and 25.
- 127. (New) The method according to claim 123, wherein n is an integer between 9 and 20.
- 128. (New) The method according to claim 123, wherein the hydrocarbon-based radical is an optionally substituted aryl or an optionally substituted alkyl.
- 129. (New) The method according to claim 118, wherein the counter-cation is an amine.
- 130. (New) The method according to claim 129, wherein the amine is a tertiary amine.
- 131. (New) The method according to claim 118, wherein the isocyanate composition comprises, on average, 1 to 5 isocyanate functions per molecule bearing isocyanate function(s).

132. (New) The method according to claim 118, wherein the isocyanate composition comprises, on average, 4/3 to 4 isocyanate functions per molecule bearing isocyanate function(s).

133. (New) The method according to claim 118, wherein the blocking agent contains at least one labile hydrogen.

- 134. (New) The method according to claim 118, wherein the blocking agent contains at least one labile hydrogen and wherein the pKa of the reactive hydrogens is at least equal to 2 but is not more than 11.
- 135. (New) The method according to claim 134, wherein the pKa of the reactive hydrogens is at least equal to 3.
- 136. (New) The method according to claim 134, wherein the pKa of the reactive hydrogens is at least equal to 5.
- 137. (New) The method according to claim 134, wherein the pKa of the reactive hydrogens is not more than 10.

Chy.

- 138. (New) The method according to claim 134, wherein the pKa of the reactive hydrogens is not more than 9.
- 139. (New) The method according to claim 134, wherein the pH of the aqueous phase is maintained at a value of not more than 12 throughout the reaction.
- 140. (New) The method according to claim 139, wherein the pH is maintained at a value of not more than 11 throughout the reaction.
- 141. (New) The method according to claim 139, wherein the pH is maintained at a value of not more than 10 throughout the reaction.
- 142. (New) The method according to claim 118, wherein the pH of the aqueous phase is maintained at a value at least equal to the value (pKa-2) throughout the reaction.
- 143. (New) The method according to claim 142, wherein the pH is maintained at a value at least equal to the value (pKa-1).
- 144. (New) The method according to claim 142, wherein the pKa is maintained at a value at least equal to the pKa value of the blocking agent, or one of the blocking agents, throughout the reaction.

Cont.

145. (New) The method according to claim 142, wherein the mass ratio between the surfactant and the isocyanates is less than 20% and greater than 2%.

- 146. (New) The method according to claim 145, wherein the mass ratio between the surfactant and the isocyanates is between 4 and 10%.
- 147. (New) A plant for carrying out the process according to claim 66, the plant comprising:
- a shear means, which can generate a shear rate (speed gradient) of greater than $1000~{\rm s}^{\text{-1}}$ and less than $1{,}000{,}000~{\rm s}^{\text{-1}}$; and
 - means for injecting an isocyanate composition into an aqueous phase.
 - 148. (New) The plant according to claim 147, wherein the shear means is a mill.
- 149. (New) The plant according to claim 148, wherein the mill is a colloidal mill.
- 150. (New) The plant according to claim 147, wherein the shear means can generate a shear rate greater than 20,000s⁻¹.

C)

151. (New) The plant according to claim 147, wherein the shear means can generate a shear rate of less than 200,000s⁻¹.

- 152. (New) The plant according to claim 147, further comprising at least one means selected from the group consisting of a means for recirculating the aqueous phase in the form of a blocked polyisocyanate emulsion, a means for regulating the injection flow rate of the isocyanate composition into the aqueous phases, a means for homogenizing the emulsion, a means for cooling the system and a means for removing the aqueous blocked polyisocyanate emulsion.
- 153. (New) The plant according to claim 147, wherein the means for injecting the polyisocyanate composition comprises:
- a vat for premixing the polyisocyanate with all or some of the surfactant or the solvent; and
 - an inlet pipe for the polyisocyanate composition in contact with the blocking agent.
- 154. (New) The plant according to claim 147, wherein the recirculation means comprises a recirculation loop.
- 155. (New) A composition comprising a blocked polyisocyanate emulsion, comprising a hydrophilic part formed of an anionic function selected from the group

tod tod

consisting of a phosphate, a phosphonate, and a phosphinate group and at least one of a polyethylene glycol chain fragment and a polypropylene glycol chain fragment, and a lipophilic part selected from the group consisting of an alkyl group and an aryl group, wherein the composition contains not more than 50%, by mass, of water relative to the emulsion.

- 156. (New) Composition according to claim 155, wherein the amount of blocked polyisocyanate(s) in the emulsion represents at least 55% by mass relative to the total mass of the emulsion.
- 157. (New) Composition according to claim 155, wherein the emulsion is formed of particles having a Sauter diameter less than or equal to 5 μ m and a dispersion width less than or equal to 5 μ m.
- 158. (New) The composition according to claim 156, comprising not more than 10%, by mass, of surfactant, relative to the mass of the isocyanate composition.

159. (New) The composition according to claim 155, wherein the surfactant corresponds to the formula:

Coup

$$(O)_{p} \xrightarrow{(X \\ (O)_{p} \\ (O)_{p}} (X \xrightarrow{(A)_{p} \\ (A)_{p} \\ (A)_{p} \\ (A)_{p} \\ (A)_{p} \xrightarrow{(A)_{p} \\ (A)_{p} \\ (A)_{p} \\ (A)_{p} \\ (A)_{p} \xrightarrow{(A)_{p} \\ (A)_{p} \xrightarrow{(A)_{p} \\ (A)_{p} \\ (A)_{p} \xrightarrow{(A)_{p} \xrightarrow{(A)_{p} \\ (A)_{p} \xrightarrow{(A)_{p} \\ (A)_{p} \xrightarrow{(A)_{p} \xrightarrow{(A)_{p} \\ (A)_{p} \xrightarrow{(A)_{p} \xrightarrow{(A)_{p} \\ (A)_{p} \xrightarrow{(A)_{p} \xrightarrow{(A)_{p} \xrightarrow{(A)_{p} \\ (A)_{p} \xrightarrow{(A)_{p} \xrightarrow{(A)_{$$

160. (New) The composition according to claim 155, wherein the surfactant corresponds to the formula:

$$(O)_{p} \xrightarrow{(X)_{S}} (X \xrightarrow{O)_{S}} (R_{2})_{q}$$

$$(O)_{p} \xrightarrow{E} (X \xrightarrow{O)_{n}} (R_{1})_{q}$$

where, when q is zero:

$$(O)_{p}^{(O)_{m}}$$

where p represents zero or an integer between 1 and 2 (closed intervals, i.e. including the limits);

where m represents zero or an integer between 1 and 2 (closed intervals, i.e. including the limits);

where the sum p+m+q is not more than three;

where the sum 1+p+2m+q is equal to three or five;

where X and X', which are similar or different, represent a divalent radical containing not more than two carbon-based chain members;

where n and s, which are similar or different, represent an integer chosen between 5 and 30 (closed intervals, i.e. including the limits); and

where R_1 and R_2 , which are similar or different, represent a hydrocarbon-based radical.

- 161. (New) The composition according to claim 160, wherein n and s represent an integer between 5 and 25.
- 162. (New) The composition according to claim 155, wherein n and s represent an integer between 9 and 20.
- 163. (New) The composition according to claim 155, having a viscosity of less than 5,500 mPa.s at, at least, 68% solids, at 25°C.

Const

164. (New) The composition according claim 155, having a viscosity of less than 1,000 mPa.s at, at least, 60% solids, at 25°C.

Cy

- 165. (New) The composition according to claim 155, further comprising a release catalyst.
- 166. (New) The composition according to claim 165, wherein the release catalyst is a latent atalyst.
- 167. (New) The composition according to claim 155, further comprising at least one polyol.
- 168. (New) The composition according to claim 167, wherein the polyol is a nanolatex whose d_{80} is not more than 1 micrometer.
- 169. (New) The composition according to claim 155, further comprising an isocyanate emulsion whose d_{80} is not more than 10 micrometers.
- 170. (New) The composition according to claim 155, wherein the water content is between 10 and 70% (oil-in-water emulsion).

Application No. <u>09/486,343</u> Attorney's Docket No. <u>004900-174</u> Page 23

171. (New) The composition according to claim 155, wherein the content of isocyanate, emulsifier and alcohol is between 30 and 70%.

My

172. (New) A process for producing coatings, the process comprising: applying the composition according to claim 155 in the form of a layer of between 20 μ m and 200 μ m in thickness; and

- heating to a temperature of at least $80^{\circ} C$.

173. (New) A coating obtained by the process according to Claim 172.